**3-(4-Methoxyphenyl)oxetane-3-sulfonyl azide (XXX)**

Oxetane sulfonyl fluoride **XX** (49 mg, 0.2 mmol, 1.0 equiv) and NaN3 (39 mg, 0.6 mmol, 3.0 equiv) were added to a flame‑dried vial and sealed. 15‑Crown‑5 ether (0.12 mL, 0.6 mmol, 3.0 equiv) and anhydrous THF (0.67 mL, 0.3 M) were added sequentially. After stirring at ambient temperature for 24 h, the reaction mixture was quenched with aq. 1 M NaOH (10 mL) and stirred for 5 min. The reaction mixture was diluted with EtOAc (10 mL), the phases separated, and the aqueous layer was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na2SO4, filtered, and concentrated *in vacuo*. Purification by automated flash column chromatography (0–50% EtOAc/hexane, see below) afforded oxetane sulfonyl azide **XXX** as a white, crystalline solid (49 mg, 90%). R*f* =0.53 (50% EtOAc/hexane); IR (film)/cm-1 2956, 2917, 2891, 2848, 2136 (N3), 1609, 1514, 1462, 1359 (SO2), 1304, 1254, 1180, 1154, 1025, 999, 837, 752, 622, 554; 1H NMR (400 MHz, CDCl3) δ 7.26 (d, *J* = 8.5 Hz, 2H, 2 × Ar­­‑CH), 7.02 (d, *J* = 8.5 Hz, 2H, 2 × Ar­­-CH), 5.40 (d, *J* = 7.4 Hz, 2H, C*H*HOC*H*H), 5.21 (d, *J* = 7.4 Hz, 2H, C*H*HOC*H*H), 3.87 (s, 3H, OCH3); 13C NMR (101 MHz, CDCl3) δ 160.9 (Ar-CqOCH3), 129.8 (2 × Ar‑CH), 124.4 (Ar‑*C*qCq), 114.6 (2 × Ar­­-CH), 77.0 (CH2OCH2), 71.6 (Cq), 55.5 (OCH3); mass ion not found by HRMS (TOF‑MS‑ES+) structure confirmed by X-ray structural analysis.

**Automated Column Conditions**: Run on a Biotage® Selekt system. Column type: Biotage® Sfär® HC 10 g. Flow rate: 40 mL/min. Sample mass: 100 mg. Solvent A: EtOAc, Solvent B: hexane. UV wavelength detection: λ 200–400 nm. See trace and gradient below. From the left: peak 1 (blue): 15‑crown‑5 ether + oxetane fluoride **XXX**, peak 2 (red): oxetane sulfonyl azide **XXX**.

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